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Laboratory Preparation of Potato Granules by Solvent Extraction

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A laboratory method is described for dehydrating mashed potatoes by extraction with organic liquids. The mashed potatoes are suspended three times in the liquid; the aqueous solvent is separated from the potato solids by filtration after each treatment. The final filter cake contains only about 5% moisture and is readily dried by mild heat. Powder prepared by this method is stable, light in color, and is easily reconstituted in water over a wide temperature range to mashed potatoes of good texture and acceptable flavor.

Research and development work on the dehydration of mashed potatoes to powder form (granules) was active in England and in Europe even prior to World War II. The British industry produced about 12 millian pounds of granules during the war. Olson and Harrington *(1)* recently reviewed the literature on granules.

Most of the dehydrated potatoes produced in the United States during World War II were blanched and diced or sliced before being dried. This product was suitable for frying or boiling but not for mashing. Production of granules for civilian consumption was started in the U. S. after the close of the war. Demand for dehydrated mashed potatoes is increasing slowly but steadily as more people become aware of the convenience that the preparation offers. Since the outbreak of fighting in Korea, interest in potato granules has increased greatly because the product has proved successful in feeding troops. Four American manufacturers are now engaged in production, and research on the subject has been stimulated.

GENERAL METHODS OF PRODUCING GRANULES

The general methods employed in producing granules consist in cooking the potatoes, mashing them to break the bonds orienting the potato cells, reducing the moisture content to a point at which the tissue can be granulated, and finally drying to 7-10% moisture content. In commercial practice, moisture content is reduced to about 35% by mixing previously dried granules with the fresh mash. Other methods of reducing the moisture content have been proposed or used commercially to a limited extent. These include partial drying, in which heat is employed under mild conditions and severe mechanical stress is avoided, and the so-called "freeze and thaw" method. In the latter process, certain changes occur during the freezing so that juice is liberated during the thawing.

METHODS OF DEHYDRATING MASHED POTATOES BY SOLVENTS

We are unaware of any previous method of dehydrating mashed potatoes by extracting the water with an organic liquid. When mashed potatoes are stirred into a water-miscible organic liquid, the solvent evidently enters the cells, where it mixes with the juice. After the liquid phase has equilibrated, the aqueous solvent is removed. The treatment is repeated until the potato cells have a low residual moisture content, after which they are dried with mild heat. Figure 1 shows photomicrographs of cells in cooked potato tissue before and after dehydration to various degree by ethyl alcohol. The effect of alcohol on gelatinized starch is to change it from a transparent paste into a white, opaque solid. As the photomicrographs show, the interior of

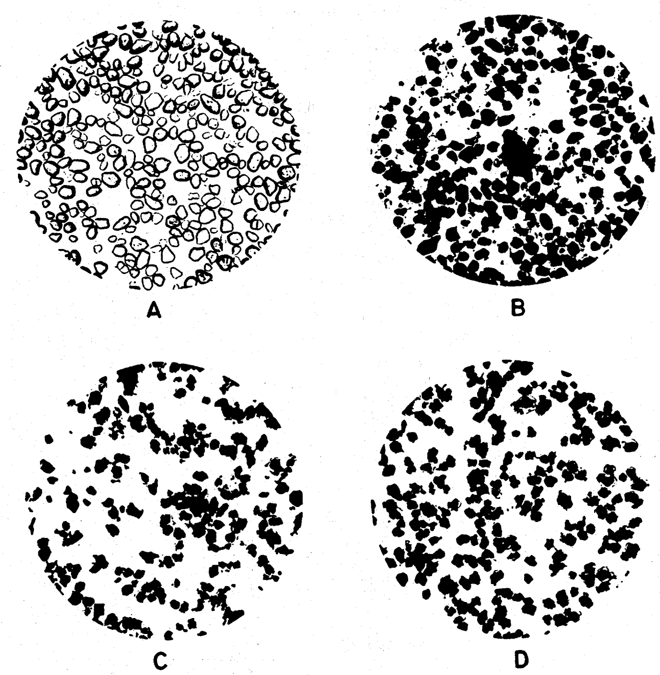

Figure 1. Photomicrographs of cooked, mashed potato tissue; magnification 12 ×: Cells mounted in (a) water; (b) 50% ethanol; (c) 70% ethanol; (d) 95% ethanol.

the potato cell becomes opaque. Although the reduction in particle size is not pronounced, some shrinkage accompanies alcoholic dehydration. Some broken cells can be seen in B, C, and D of Figure 1. Ruptured cells must be kept at a minimum because the presence of appreciable "free" starch in mashed potatoes leads to pastiness.

An organic solvent such as alcohol firms potato cells. It also provides a medium to keep the cells separated until dehydration is advanced and stickiness gone. In its ideal form, the final product consists essentially of discrete potato cells with a minimum of agglomerates. This is an important attribute of the solvent method, because the granulation step, designed to reduce moist mashed potato powder to substantially unicellular form, is one of the most difficult in the production of granules by thermal dehydration.

Several methods, including vacuum filtration and centrifugation, have been employed in separating the aqueous solvent from the potato solids.

Continuous liquid extraction and vapor-phase extraction of mashed potato tissue were explored briefly and abandoned for various reasons. Passage of the liquid solvent through a vertical column is slow, particularly in the early stage of dehydration. Vapor-phase dehydration was unsuccessful because the vaporized organic liquid tended to pass through in channels.

The following procedure has been found convenient in the laboratory for dehydrating potatoes by solvent extraction. The potatoes are generally not peeled unless the raw material has an unusually tough, heavy skin. The skin is screened off along

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with the coarse material after the product is dried to a powder. Clean potatoes are sliced into strips about ¾" thick and steamed for 35 minutes. The potatoes are then mashed by beating for 2 minutes at high speed in a Hobart Mixer (Model N-50),[b] with the flat beater attachment. The organic solvent is added while the beater moves at slow speed. The slurry is filtered after 15 minutes of mixing. The filter cake is broken up and resuspended in fresh solvent if more than one extractive step is to be employed.

A number of water-miscible solvents were evaluated. The more promising ones were tried at different ratios of solvent: potato to compare their effectiveness in removing water and to obtain information on the amount of solids removed. Data on 5 solvents are summarized in Table 1. These are results of a single extraction; a maximum of 2 parts by weight of solvent to 1 of wet mashed potatoes was used. Not enough water is removed under such conditions to permit drying the product to a powder.

TABLE 1

One-step extraction of mashed potatoes with different solvents at various ratios

Part anhyd. solvent/part potato[c, d]	Water extracted, % of total	Parts water extracted/ part solvent[c]	Solids dissolved, % of total	Solids extracted, % of total
		Methanol		
2	82.3	0.32	14.7	12.2
1.5	81.9	0.43	15.1	12.3
1	74.3	0.58	16.0	11.9
0.5	65.7	1.04	18.2	12.0
0.25	41.9	1.32	18.3	7.7
		Acetone		
2	86.7	0.35	10.9	9.3
1.5	83.7	0.45	11.1	9.4
1	76.7	0.61	13.1	10.1
0.5	73.8	1.18	16.8	12.6
0.25	49.5	1.57	21.4	10.5
		Ethanol		
2	81.2	0.33	16.8	13.6
1.5	79.2	0.42	18.3	14.5
1	77.6	0.62	19.8	15.4
0.5	71.4	1.14	23.9	17.5
0.25	24.9	0.80	33.8	8.4
		Isopropanol		
2	79.5	0.33	11.3	9.3
1.5	83.1	0.44	12.1	10.1
1	80.5	0.64	12.9	10.4
0.5	68.2	1.10	17.4	12.0
0.25	29.9	0.95	24.2	7.2
		Tertiary Butanol		
2	84.0	0.34	11.9	10.0
1.5	80.0	0.43	12.9	10.2
1	76.4	0.61	14.9	11.4
0.5	60.2	0.96	22.7	13.8
0.25	49.5	1.58	28.7	14.2

[c] By weight.
[d] Solvent concentrations in liquid phase of solvent-potato mixtures are as follows for the various ratios of solvent to potato: ratio of 2 parts solvent to 1 of potato, 71.7%; 1.5 to 1, 65.5%; 1 to 1, 55.8%; 0.5 to 1, 38.8%; 0.25 to 1, 24.1%.

The amount of dissolved solids increased in each case as the proportion of water to organic solvent was increased; i.e., with decreasing ratio of solvent to potato. Since the suspensions were centrifuged after mixing and standing, data on the extracted solids actually show amounts of solubles separated in the liquid phase from the settled layer. The settled layer became less compact and contained more liquid when less organic solvent was present.

The combination of sugars, inorganic salts, organic acids, and nitrogen compounds—those compounds in uncooked potato that are soluble in water—ordinarily constitute about 20% of the total solids. It will be noted in Table 1 that the amount of dissolved solids exceeded 20% in some instances where the concentration of solvent was low. Dissolved starch contributed to these unusually high values. The presence of soluble starch in the centrifugate was proved by the iodine test.

Several factors are involved in arriving at the preferred solvent or solvents. Acetone removes water efficiently while extracting relatively little of the potato solids. It distills in anhydrous form (advantageous for re-use of the solvent) at a low boiling point. However, acetone imparts disagreeable flavor and odor to the product. Attempts are being made to correct this. Methanol is reasonably good in water extraction and distills in anhydrous form. Ethanol is relatively good as an extractive solvent, particularly at the intermediate concentrations shown in Table 1, and yields a final product of good flavor. The 5% water present in distilled ethanol limits its dehydrating value. Isopropanol and tertiary butanol were given little consideration, because their azeotropes contain considerable water.

Single vs. several extractions. A comparison was made to determine the relative efficiency and possible effect on texture and flavor of several extractions of the mashed potatoes.

A better yield of fine particles (Table 2), dependent largely on the concentration of organic liquid in contact with the potato solids before the final filtration, was obtained with multiple extractions. Not only a higher yield of fine powder was obtained, but less solvent was needed. A large percentage of fine material is necessary to give a product that reconstitutes to a smooth, lump-free mash.

TABLE 2

Effect of number of extractions on granulating efficiency. (Extraction of 1000 g. of mashed potatoes)

Solvent,[e] grams/treatment			Solvent in filtrate, %	Yield of dry granules, % of total yield	
1st	2d	3d		—30 mesh	—80 mesh
			Ethanol		
3000	....[f]	....[f]	71	60	50
2000	1000	....[f]	88[g]	93	82
			Methanol		
2000	....[f]	....[f]	70	54	18
1000	500	....[f]	83[g]	75	55
			Ethanol		
1500	1000	....[f]	85[g]	88	70
500	400	300	84[g]	86	78

[e] Ethanol, 95%; methanol, anhydrous.
[f] No treatment.
[g] Final filtrate, before drying.

Three-step procedure for extraction. As indicated by the data in Table 2, a 3-step extraction offers considerably more economy in solvent requirement than a 2-step or single extraction. Hence a similar procedure has been tentatively adopted as our standard laboratory method. The first extraction is carried out with the aqueous solvent obtained by combining the second and third filtrates of the previous batch. (At the beginning, before filtrates are available from previous batches, the first extraction is made with 500 g. of distilled solvent, which was 95% in the case of ethanol and anhydrous with acetone and methanol). The second and third extractions are made with fresh solvent distilled from the filtrate of the first extraction.

One thousand grams of mashed potatoes are suspended in organic solvent in accordance with the directions previously given. The suspension is filtered through Schleicher and Schüll Co. "Shark Skin" paper on a 24-cm. diameter Buchner funnel with suction. The acetone suspension filters the most rapidly, and the ethanol suspension the most slowly. The filtration rate of the methanol suspension is intermediate. The filter cake is broken up and suspended in 400 g. of fresh solvent. After being mixed for about 15 minutes, the suspension is again filtered on a Buchner funnel. This filter cake is suspended in 300 g. of fresh solvent, again mixed, and filtered. The final filter cake (containing only about 5% moisture) is broken up by gentle agitation and dried to about 7% moisture content by application of mild heat.

[b] Mention of brand or trade names in this paper does not imply that the article is recommended or endorsed by the U. S. Department of Agriculture over similar articles not mentioned.

Table 3 gives data on the 3-step extraction with acetone. The figures represent average, typical values obtained after 10 or more cycles of granule preparation.

TABLE 3

Dehydration of mashed potatoes by 3-step acetone extraction. [1000 g. potatoes (19.6% total solids)]

	First suspension	Second suspension	Third suspension
Wt. of acetone, g.	830[h] (82%)	400 (anhyd.)	300 (anhyd.)
	First filtrate	Second filtrate	Third filtrate
Wt. of filtrate, g.	1400[i]	530	300
Wt. dissolved solids in filtrate, g.	25	....[j]	....
Composition filtrate, % [k]			
Acetone	42	76	90
Water	58	24	10
Composition filter cake, %			
Potato solids	37	51	54
Acetone	26	37	41
Water	37	12	5
Wt. filter cake, g.	467	338	316

[h] Combination of second and third filtrates of preceding cycle.
[i] Filtrate goes to still for recovery of anhydrous solvent.
[j] Dissolved solids determined only in first filtrate, which contains more than 95% of total solubles extracted.
[k] Liquid constituents only, not including dissolved solids.

For every 1000 g. of mashed potatoes dehydrated, 700 g. of recovered solvent are used, and approximately 1400 g. of aqueous solvent are made available for redistillation. As shown in Table 3, the 1400 g. of 42% acetone will yield 588 g. of anhydrous solvent, and nearly 130 g. additional are available in the final filter cake.

About 50% of the water-soluble potato solids (approximately 10% of the total solids) are removed in solvent dehydration. These solubles can be recovered by distilling off the water, under reduced pressure, from the solution remaining after recovery of the organic solvent. When the solution becomes sirupy, containing about 50% solids, evaporation of water is stopped, and the sirup is mixed with an equal weight of dry granules to prepare a free-flowing flavor concentrate that mixes readily with mashed potato powder.

Nature of the extracted solubles. The mixture of substances extracted from cooked potatoes by aqueous organic solvents is a brown material, waxy when water-free. No complete analaysis has been made of this mixture. An aqueous alcoholic solution obtained by a 2-step extraction of Maine Green Mountain potatoes contained 5.6% nitrogen (35% nitrogen compounds calculated by the 6.25 conversion factor) and inorganic substances equivalent to 20.0% ash, on the dry basis. Undoubtedly, products of the reaction between amino compounds and reducing sugars were present, and presumably sugars, organic acids, minor constituents, and some fat.

COMPARISON OF PROPERTIES OF SOLVENT- AND THERMAL-DEHYDRATED POTATOES

Density. The density (apparent) of mashed potato powder produced by the solvent method ("solvent granules") is lower than that of the commercial, thermal-dehydrated product ("thermal granules"). Powder density varies with the solvent used. With the 3 solvents most commonly used, density decreased with decreasing boiling point. Thus in comparative experiments, ethanol boiling at 78.4° C. produced granules having a density of 0.78; methanol boiling at 64.7° C. gave a density of 0.73; and acetone boiling at 56.5° C. produced a density of 0.67. Thermal granules typically have a density of about 0.85.

Water absorption. Solvent granules can be reconstituted in water ranging from cold to near boiling. Thermal granules, however, absorb little water below about 140° F. and require 160-180° for best results. All granules tend to give a pasty product when reconstituted in boiling water.

Color. Solvent granules are nearly white. Some yellow color is imparted by putting back extracted solubles, but the product is still lighter than thermal granules.

Flavor. Solvent granules reconstitute to a mash of bland flavor when considerably less than the normal amount of potato solubles is present. Replacement of solubles can give a reconstituted product similar in flavor to fresh mash. When solvent granules are dried with more than the usual amount of heat treatment, the product more closely resembles thermal granules in flavor.

Storage properties. Comparative storage tests on solvent and thermal granules are in progress. Solvent granules apparently need no sulfite treatment to prevent deterioration of flavor and color in storage. Sulfiting of thermal granules, though, has been a common practice in the industry.

Yield of product. From 1000 parts of potatoes (20% total solids), a yield of 176-178 parts of granules at 7% moisture is obtained by dehydration with ethanol or acetone without "add back" of solubles. Twenty-five to 30 parts of potato solubles remain in the spent filtrate, which is distilled to recover solvent. Hence, mechanical losses are slight in laboratory practice. Typically, 86-88% of the granules pass through a 30-mesh screen and 76-78% through an 80-mesh screen. The material retained on a 30-mesh screen consists largely of small bits of skin and skin lumped with potato tissue.

COMPOSITION OF SOLVENT GRANULES

Composition of granules prepared by a given process depends on the nature of the solvent and the character of the raw material. Potatoes representing 3 varieties were extracted with various solvents. Variety had a pronounced effect. For example, in extraction with alcohol only 15% of the total nitrogen was removed from Florida Sebago, 35% from the Maine Green Mountain variety, and 38% from North Carolina Cobbler potatoes. Different amounts of fat, sugars, inorganic substances, and organic acids were extracted from different varieties of potatoes by the same solvent. The different solvents produced appreciably different nitrogen retention in the same variety. There was relatively high nitrogen retention when acetone and ethanol were used. Acetone extracted less of the sugars and inorganic substances than ethanol and much less than methanol. Table 4 shows the results with the Florida Sebago variety. Results obtained with the Green Mountain and North Carolina Cobbler varieties were similar to those with the Florida Sebago.

TABLE 4

Changes in composition produced by solvent extraction of mashed potatoes (Florida Sebago)

Constituent, % [1]	In raw material	Granules prepared by use of Ethanol	Methanol	Acetone
Total N	1.48	1.26	1.05	1.31
Crude fat	0.66	0.33	0.39	0.41
Total sugars	1.98	0.69	0.46	1.27
Starch	73.4	78.1	82.6	77.5
Ash	5.71	2.36	1.73	2.46

[1] Moisture-free basis.

FLAVOR OF SOLVENT GRANULES AND ITS RELATION TO THE AMOUNTS OF SOLUBLES AND RESIDUAL SOLVENT PRESENT

Tasters in this laboratory have repeatedly compared fresh mashed potatoes with reconstituted, commercial thermal granules and with experimentally produced solvent granules. No reconstituted dehydrated product is generally considered equal to the fresh mashed potatoes. Preference concerning thermal and solvent granules is largely a matter of individual opinion. Prominent factors in forming an opinion are preferences with regard to bland vs. pronounced potato flavor and white vs. slightly yellow color.

Because they have their original content of flavorful solubles, thermal granules have more potato flavor than solvent preparations. In addition, thermal granules have a flavor quality presumably imparted by the heat treatment. The sulfite content of commercial thermal granules is apparently too low to be objectionable to most persons.

Tasters were in almost unanimous agreement that "add back" of solubles improves the flavor of solvent granules. Beyond 80% of the original solubles content, however, they could detect no further improvement as the flavorful substances were increased to their full complement.

Dehydrated mashed potatoes retain a small amount of organic liquid that is held rather tenaciously during drying. It is doubtful if as much as 0.5 to 1% of acetone or alcohol in the dry powder is detectable by taste after reconstitution. Obviously, though, the residual solvent must be reduced to virtually zero in a food product. Indications are that drying to about 7% moisture followed by remoistening to 10-12% will remove the adsorbed solvent. As an example, samples of granules containing 2.2% methanol and 1.6% acetone were subjected to light steaming for 35 minutes and then redried to 7% moisture content. After this, the former had 0.09% methanol and the latter 0.01% acetone content.

A more direct method is being sought for removing adsorbed solvent during drying. Slow drying at relatively low temperature is promising in this regard. The data in Table 5 show how reduction of the drying temperature resulted in pronounced lowering of the residual ethanol content. There are indications that residual solvent content can be reduced still more by letting the mashed potatoes cool to room temperature before suspending them in the solvent, instead of using warm potatoes, which was our usual procedure. This is certainly true with ethanol. Residual alcohol in the final product can be reduced one-third to one-half by letting the mash stand for 4 hours at room temperature before starting the extraction.

Engineering studies under way at this laboratory are designed to aid in selecting equipment and to determine procedures for larger scale production. One major objective is to devise methods for handling the slurries and drying the final filter cake under conditions that will minimize loss of solvent. Another objective is to provide a cost estimate for production of solvent granules.

TABLE 5

Effect of drying temperature on residual content of ethanol in granules (2 hrs. in tray drier)

Temperature, °F.	Final moisture content, %	Residual ethanol in granules, % [m]
92	11.6	0.6
108	7.6	0.8
130	7.1	0.9
150	5.9	1.1
170	3.3	1.2
190	2.1	1.1

[m] Moisture-free basis.

SUMMARY AND CONCLUSIONS

Laboratory studies have shown that granules of good color, texture, and acceptable flavor can be prepared by extracting the water from potatoes with certain organic liquids. Of the solvents tried, ethanol, methanol, and acetone gave the best results. Acetone extracted the least amount of water solubles, but granules prepared by alcohol extraction were superior in flavor.

A 3-step laboratory procedure, each step consisting of suspending mashed potatoes in the solvent and filtering off the liquid phase, has been employed. Equilibrium conditions are established after several cycles. The combination of filtrates from the second and third treatments of the preceding cycle is used for the first extraction of wet mash. Hence, the solvent is used twice before recovery by distillation. Distilled solvent recovered from spent aqueous filtrate, plus fresh solvent to replace that lost in drying the final cake, is used for the second and third extractions. In commercial manufacture of granules, however, solvent from the filter cake would necessarily be recovered. Because of the promise indicated in this preliminary work, laboratory studies on solvent granules are being continued.

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